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PROCEDURE FOR OXYGEN INDEX TESTS AT THE CANADIAN EXPLOSIVE ATMOSPHERES LABORATORY

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by

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ABSTRACT

A statistically valid procedure is proposed for testing the oxygen index (a measure of flammability) of samples submitted to the Canadian Explosive Atmospheres Laboratory (CEAL) as "flame-resistant" materials used in underground mines. The limitations of the current apparatus are also discussed. The oxygen index of conveyor belt samples were measured as a function of sample width: the graphs are S-shaped. For consistent results, it is recommended that samples be 25 mm by the full thickness.

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MARCHE À SUIVRE LORS DES ESSAIS D'INDICE D'OXYGÈNE AU LABORATOIRE CANADIEN SUR LES ATMOSPHÈRES EXPLOSIVES

par

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RÉSUMÉ

On propose une marche à suivre statistiquement acceptable pour faire des essais d'indice d'oxygène (mesure de l'inflammabilité) sur des échantillons fournis au Laboratoire canadien sur les atmosphères explosives (LCAE) utilisés comme matériau ignifuge dans les mines sous-terraines. On discute aussi des limites de l'équipement concerné.

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INTRODUCTION

The oxygen index test (sometimes referred to as the "critical oxygen index" or "limiting oxygen index" test) is used widely as a measure of the flammability of materials, particularly plastics. The oxygen index is defined as the percentage of oxygen in an oxygen/nitrogen mixture that will just support flaming combustion. As with all small-scale tests on flammability, it is a subject of controversy on its applicability to real-life fires. The purpose of the present work is to establish a standard procedure so that meaningful correlations with other methods can be undertaken.

APPARATUS

The United States Testing Company's, Model 8100 Oxygen Index Tester was purchased by CEAL. A picture of it, as supplied, is shown in Fig. 1.

According to the manufacturer, the apparatus meets the technical requirements of ASTM method D2863-77. This claim appears to be overstated. The ASTM requires that the gas flows be monitored within 1% in the range being used. The flowmeters used in model 8100 are calibrated to ±1% of <u>full scale</u> according to the manufacturer. For a typical fire-resistant material of O.I. = 30%, the oxygen flow rate required would be 55 $cm^3 s^{-1}$; compared to the flowmeter range of 13 to 136 cm^3/s; the nitrogen flow rate would be 127 cm^3/s compared to the flowmeter range of 16 to 178 cm^3/s. Hence, the uncertainties in the flow rates are 2.5% and 1.4%, respectively. These uncertainties result in an uncertainty of up to 1.1 in the O.I. at O.I. = 30 and up to 0.8 at O.I. = 20. If the gas flowsystems were constructed up to ASTM standard, the resultant uncertainty in the O.I. would be only ±0.4.

According to the ASTM, the standard deviation in O.I. <u>within</u> a laboratory ranges from 0.1 for clean-burning materials to 1.0 for erratic materials. Comparing laboratory-to-laboratory results, the standard deviation in O.I. ranges from 0.4 to 1.5. These larger values on interlaboratory results may just be due to the ±1% tolerance allowed by ASTM on the flow rates.

In view of the limited accuracy of the flowmeters in our apparatus, we cannot expect to approach the lower values of the standard deviation. However, most of the materials tested at CEAL will be composites; hence, will have intrinsically larger standard deviations. That is to say, although the apparatus is not up to ASTM standards, it should satisfy the needs of CEAL.

The precision of setting each flowmeter is, if done carefully, ca. 0.5 cm^3/s, producing an uncertainty in the oxygen index value of 0.3. The precision is useful for comparing O.I. values done on the same apparatus, but for comparing values obtained elsewhere, the accuracy + precision must be used.

Entrainment of ambient air into the column has been found to affect the accuracy of O.I. tests (1). A drawing of a brass annulus designed to fit onto the top of the glass column and eliminate the problem has been published (2). A copy of this brass annulus was constructed by the Technical Services Division and is now in routine use.

A rod of polymethylmethacrylate, which has a well-defined O.I. of 17.3%, is often used as a standard for O.I. apparatus. Our apparatus yielded a value of 18.0%, with a standard deviation of about 0.5%. This error in O.I. is within the estimated tolerance of our apparatus (= precision + calibration accuracy = 0.3 + 0.8 = 1.1%).

A detailed procedure is given in Appendix A.

STATISTICAL APPROACH

The ASTM method defines the O.I. as the minimum concentration of oxygen that will just support flaming combustion. For materials that have a very sharp cutoff between burning and not burning (i.e., 100% burns at one concentration and 0% burns at the experimentally next lower concentration), such a definition will not cause difficulties. On the other hand, most materials will not have such a sharp cutoff: at several concentrations, the probability of burning will have values between 0 and 100%. (The curve of probability versus concentration should follow the integral form of the normal distribution.) In that case, one must define the O.I. to be that concentration of O_2 at which the probability of burning has a specific value. The 50% point requires the least number of tests to determine its value to a given accuracy. Therefore, it is recommended that the percentage of O_2 at which 50% of the samples burn be defined as the O.I.

W. Dixon has designed a modified Bruceton up-and-down method, which allows an accurate determination of the 50% point by testing only a few samples (3). His table (attached) is self-explanatory. Note that the test levels must be equidistant.

This statistical approach assumes that the interval between test levels is roughly σ (the standard deviation). The empirical σ can not be less than 0.3, because that is the precision of the apparatus. Our tests on conveyor belt samples indicate that σ is roughly 0.5% O_2. Therefore, unless the sample shows extremely erratic behaviour, it is recommended that a standard interval size of 0.5% be used.

Based on the foregoing, the following procedures are recommended.

(1) Untested material

Start at an appropriate value (e.g., 28% for a flame-resistant material). If the sample burns, decrease the $\%O_2$ by 2%; otherwise, increase it by 2%. Continue until the opposite result occurs. Then test five more samples using an increment of 0.5%. Use Table 1 to calculate the O.I., using only the last six tests for the calculations.

(2) Repeat material (e.g., Quality Assurance)

Prepare six samples. Start the testing at the previously determined O.I. Use increments of 0.5%, test all samples. Use Table 1 to calculate the O.I. If this value differs from the previous by less than 1.0, then the two materials can be considered to be equivalent in flame-resistency. Otherwise, another set of six tests are required. If the same result is obtained, there is a strong likelihood that the two materials are not the same.

If the original O.I. tests had been carried out elsewhere, a difference of up to 2.0% would have to be accepted.

A worksheet for the O.I. test is attached (Appendix B).

CONVEYOR BELT TESTS

The first question that must be answered is the dimensions of the test sample. A European study (4) found that the O.I. increased with increasing sample width and better reproducibility was obtained with 60 mm wide samples than with 3 and 30 mm wide samples. The ISO recommends 50 mm wide by full thickness. The Australians (private communication) use 6.5 mm wide by full thickness (which is based on the type A sample of ASTM D2863-77, except the latter uses 3.0 mm thick samples).

The O.I. of three types of belts (PVC flame-resistant, rubber flame-resistant, rubber non-flame resistant) were measured as a function of sample width (Fig. 2). All three belts show a distinct S-shaped curve; the position of the steep section appears to be a function of the belt type. During the burning of the narrow samples, the various layers tend to separate with subsequent breaking off of small pieces. This produces an increased exposed area which means that the sample can burn at a lower concentration of oxygen. The wider samples retain their integrity during burning.

A somewhat similar increase in O.I. with sample thickness has been observed for thermoplastic materials which do not burn cleanly (5). In those cases, the cause was attributed to either heat carried away by melting (of the thicker samples) or char formation (again in the thicker samples) which insulates the material from the flame. The latter mechanism may be contributing in the conveyor belt tests.

As can be seen from Fig. 2, misleading results can be obtained from a poor choice of sample width. If 5-10 mm is used (as the Australians do), then the two flame-resistant belts would be considered equivalent, which clearly is not true. Even for quality assurance, using a sample width on the sloping part of the curve would be undesirable, since it would require very careful attention in cutting the sample to the exact width. Any width above 15 mm appears to be acceptable. A very wide sample, 50-60 mm, may be affected by its proximity to the walls, as well as generating excessive quantities of heat and smoke, which would slow down testing. Therefore, it is recommended that 25 mm by the full thickness be used as the standard sample size.

Wharton has made a comprehensive review of comparisons between O.I. tests and other flammability tests reported in the literature (6). Good correlation was found between the O.I. and the Monsanto 2 ft tunnel and Swedish box test, but poorer correlation with many other small scale tests. Several studies have shown that the O.I. increases steadily with increasing concentration of fire-retardant, a useful characteristic for quality assurance work.

CONCLUSIONS

Work is currently underway at CEAL to correlate the O.I. with a large scale fire test (the propane gallery test). It is hoped that the O.I. test (possibly in combination with another small-scale test) will provide a reasonably reliable predictor of performance in the large-scale test. Apart from that, the O.I. test appears to be the best choice at the present time for quality assurance testing by CEAL.

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APPENDIX A

PROCEDURE FOR OXYGEN INDEX TESTS

1. Cut the sample to the appropriate size - for conveyor belts, normally 25 mm wide x 60-100 mm long.

2. Mark a line 50 mm from the top.

3. Clamp the sample in place.

4. Ensure that the glass column is clean and close to room temperature.

5. Place the brass annulus on top of the glass column.

6. Adjust the oxygen and nitrogen flow rates; purge for at least 30 sec.

7. Ignite the top of the sample, ensuring that the complete top is burning when the ignitor is removed.

8. If a flame, however small, lasts more than 3:00 min, or any part of the flame front reaches the 50 mm line, then consider it as a "burn".

C.E.A.L. OXYGEN INDEX TEST

Date: Operator:

Sample: Witnesses:

Manufacturer: Sample Size:

Conditioning:

Mark X if sample burns more than 3 min. or 50 mm: decrease % O_2

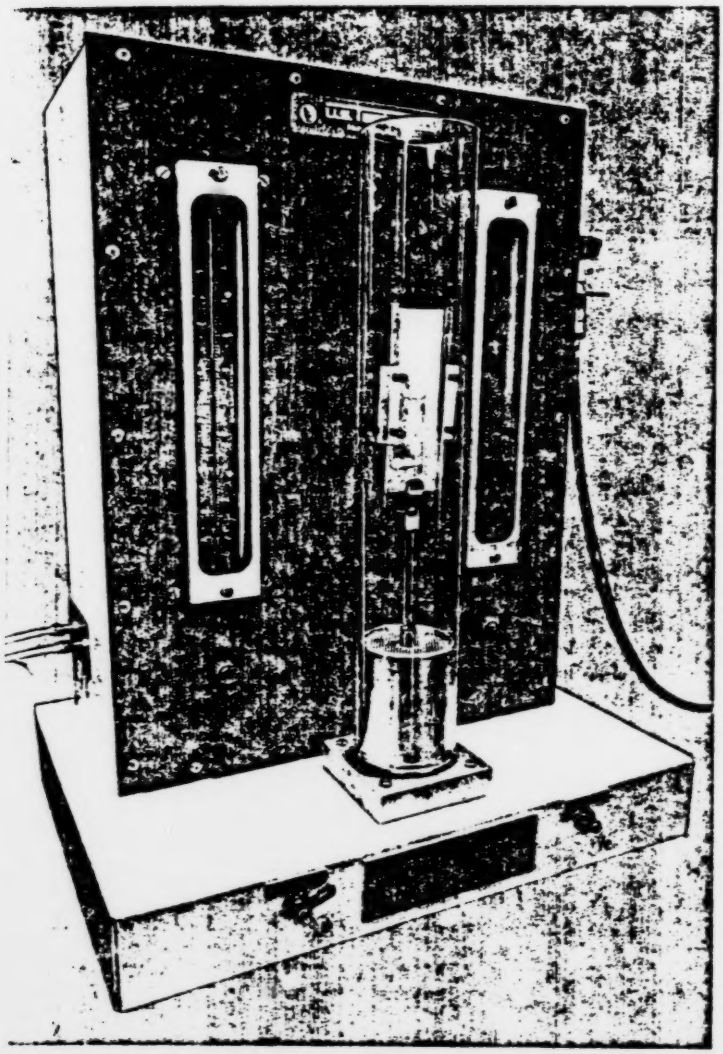
Mark actual burn time otherwise : increase % O_2

% O_2	1 2	3 4	5 6	7 8
33 32.5				
32 31.5				
30 29.5				
29 28.5				
28 27.5				
27 26.5				
26 25.5				
25 24.5				
24 23.5				
23 22.5				
22 21.5				
21				

O.I. (50% Level) =

Comments:

united states testing co., inc.



Oxygen Index Flammability Tester

Model 8100

Application:

The apparatus is designed for determining the minimum concentration of oxygen in a flowing oxygen/nitrogen mixture that will just support flaming combustion of a material. It meets the technical requirements of ASTM D-2863 for testing plastic materials, including film and cellular polymers. The Oxygen Index Flammability Tester can also be used to determine the relative flammability of textiles, paper, and other combustible materials in a laboratory environment.

Features:

- Flush-mounted, full-view oxygen and nitrogen flowmeters (calibrated to 1-percent accuracy)
  - Dual floats and dual direct reading scales showing gas flow rates in cc/sec
  - Integral flow controllers to maintain constant flow rates independent of gas inlet pressures
  - Needle valves for fine flow-rate adjustment; flow shutoff valves
  - Gas line filters
- Test column with gas mixing chamber, specimen holder, and heat-resistant glass chimney
- Specimen igniter assembly with needle valve to control flame length

General Description:

The specimen is held in a vertical position inside the glass-enclosed test column. Gases enter the test column through the metal base, which contains a bed of glass beads to evenly mix and distribute the oxygen and nitrogen. When the flow of rising gases has stabilized in the column, the specimen is ignited and observed for intensity of burning. When flaming ceases, the specimen is replaced by a fresh sample, the oxygen concentration is adjusted based on the initial test results, and the procedure is repeated. Testing continues until the concentration of oxygen required to just support flaming combustion is determined. This value is the oxygen index of the test material

The Oxygen Index Flammability Tester yields a numerical rating of fire resistance that is accurate, precise, and reproducible. The apparatus can be used as a research and development tool to evaluate fire retardant formulations or to study the effect of changes in a material on flammability. It can also be used for specification testing to assure quality control of standard materials.

Fig. 1 - Oxygen index apparatus

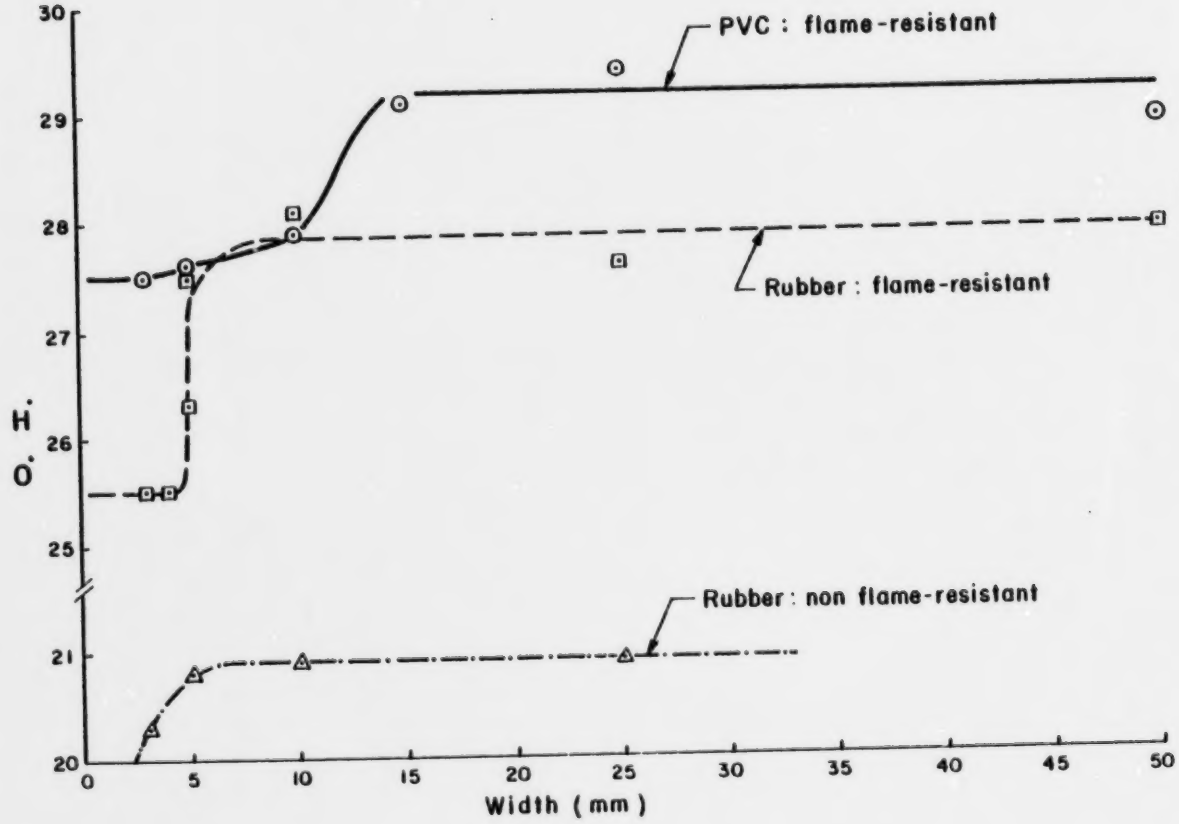


Fig. 2 - Oxygen index: effect of sample width

TABLE 1. VALUES OF k FOR ESTIMATING LD_{50} FROM UP-AND-DOWN SEQUENCE OF TRIALS OF NOMINAL LENGTH N. THE ESTIMATE OF LD_{50} IS $z_f + kd$ WHERE z_f IS THE FINAL TEST LEVEL AND d IS THE INTERVAL BETWEEN DOSE LEVELS. IF THE TABLE IS ENTERED FROM THE FOOT, THE SIGN OF k IS TO BE REVERSED

N	Second Part of Series	k for Test Series Whose First Part is					Standard Error of LD_{50}
		O	*OO*	*OOO*	*OOOO*		
2	*X*	−.500	−.388	−.378	−.377	*O*	.88σ
3	*XO*	.842	.800	.894	.894	*OX*	.76σ
	XX	−.178	.000	.026	.028	*OO*	
4	*XOO*	.299	.314	.315	.315	*OXX*	.67σ
	XOX	−.500	−.439	−.432	−.432	*OXO*	
	XXO	1.000	1.122	1.139	1.140	*OOX*	
	XXX	.194	.449	.500	.506	*OOO*	
5	*XOOO*	−.157	−.154	−.154	−.154	*OXXX*	.61σ
	XOOX	−.878	−.861	−.860	−.860	*OXXO*	
	XOXO	.701	.737	.741	.741	*OXOX*	
	XOXX	.084	.169	.181	.182	*OXOO*	
	XXOO	.305	.372	.380	.381	*OOXX*	
	XXOX	−.305	−.169	−.144	−.142	*OOXO*	
	XXXO	1.288	1.500	1.544	1.549	*OOOX*	
	XXXX	.555	.897	.985	1.000[+1]	*OOOO*	
6	*XOOOO*	−.547	−.547	−.547	−.547	*OXXXX*	.56σ
	XOOOX	−1.250	−1.247	−1.246	−1.246	*OXXXO*	
	XOOXO	.372	.380	.381	.381	*OXXOX*	
	XOOXX	−.160	−.144	−.142	−.142	*OXXOO*	
	XOXOO	.022	.039	.040	.040	*OXOXX*	
	XOXOX	−.500	−.458	−.453	−.453	*OXOXO*	
	XOXXO	1.160	1.237	1.247	1.248	*OXOOX*	
	XOXXX	.611	.732	.756	.758	*OXOOO*	
	XXOOO	−.296	−.266	−.263	−.263	*OOXXX*	
	XXOOX	−.831	−.763	−.753	−.752	*OOXXO*	
	XXOXO	.831	.935	.952	.954	*OOXOX*	
	XXOXX	.296	.463	.500	.504[+1]	*OOXOO*	
	XXXOO	.500	.648	.678	.681	*OOOXX*	
	XXXOX	−.043	.187	.244	.252[+1]	*OOOXO*	
	XXXXO	1.603	1.917	2.000	2.014[+1]	*OOOOX*	
	XXXXX	.893	1.329	1.465	1.496[+1]	*OOOOO*	
		X	*XX*	*XXX*	*XXXX*	Second Part of Series	
		−k for Series Whose First Part is					

50% level = last level tested + k x step size

EXAMPLE

```
32                X
31.5        X   O
31        O   O
```

First part of series(i.e. the tests done before there is a reversal in response) is O ; second part of series is XOOX.
50% level = 32 + (-.878)(0.5) = 31.6